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DEPARTMENT OF COMMERCE

BUREAU OF STANDARDS

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UNITED STATES GOVERNMENT MASTER SPECIFICATION FOR
PUTTY

FEDERAL SPECIFICATIONS BOARD SPECIFICATION No. 283

This specification was officially promulgated by the Federal Specifications Board on May 8, 1925, for the use of the Departments and Independent Establishments of the Government in the purchase of putty.

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I. CLASSES

The putty shall be of two classes—either whiting putty or white lead-whiting putty, as called for in the proposal.

II. MATERIAL

No details specified.

III. GENERAL REQUIREMENTS

No details specified.

IV. DETAIL REQUIREMENTS

1. PIGMENT

The pigment in whiting putty shall consist of finely powdered natural chalk of high-grade quality, with the minimum amount of pure tinting colors to produce the desired colors. It shall be free from grit, shall be practically neutral, shall possess the property of mixing with linseed oil to form good putty, and shall have the structure and other physical characteristics of the best natural whiting suitable for putty making. The total pigment shall contain not less than 95 per cent of calcium carbonate.

The pigment in white lead-whiting putty shall contain not less than 10 per cent of white lead (basic carbonate or basic sulphate), a minimum amount of pure tinting colors if necessary, the remainder to be the above-described natural whiting. The sum of the white lead and calcium carbonate in the total pigment shall be not less than 95 per cent.

2. LIQUID

The liquid in either class shall be entirely pure raw linseed oil.

3. PUTTY

The putty shall be made by thoroughly grinding the specified class of dry pigment with pure raw linseed oil to a doughlike paste of proper putty consistency that shall be smooth, uniform, and suitable, as received, for glazing purposes. The putty as received shall not be caked in the container. It shall possess the characteristic properties of and shall be equal to the best grade of putty. The color when specified shall match a sample mutually agreed upon by buyer and seller.

The putty shall consist of:

Ingredients	Maximum	Minimum
	<i>Per cent</i>	<i>Per cent</i>
Pigment.....	87	82
Pure raw linseed oil.....	18	13
Moisture and other volatile matter.....	0.5	-----
Alkali.....	Trace.	-----
Coarse particles and "skins" (total residue retained on No. 325 sieve based on pigment).....	3.0	-----

V. METHODS FOR SAMPLING AND TESTING

Deliveries will, in general, be sampled and tested by the following methods, but the purchaser reserves the right to use any additional available information to ascertain whether the material meets the specification.

1. SAMPLING

It is mutually agreed by buyer and seller that a single package out of each lot of not more than 1,000 packages shall be taken as representative of the whole. Whenever possible, an original unopened container shall be sent to the laboratory, and when this is for any reason not done the inspector shall determine by kneading with his hand whether the material has the consistency of good putty. He shall then thoroughly mix the contents of the container and draw a sample of not less than 1 pound of the thoroughly mixed putty, place it in a clean, dry metal or glass container, which must be filled with the sample, closed with a tight cover, sealed, marked, and sent to the laboratory for test with the inspector's report on caking in container.

When requested, a duplicate sample may be taken from the same package and delivered to the seller, and the inspector may take a third sample to hold for test in case of dispute.

2. LABORATORY EXAMINATION

(a) **CAKING IN CONTAINER.**—When an original package is received in the laboratory, it shall be weighed, opened, and mixed with a stiff spatula or paddle. The putty must be no more difficult to mix and knead with the hand than a normal good grade of putty. The putty shall finally be thoroughly mixed, removed from the container, and the container wiped clean and weighed. This weight subtracted from the weight of the original package gives the net weight of the contents. A portion of thoroughly mixed putty shall be placed in a clean container and the portions for the remaining tests promptly weighed out.

(b) **MOISTURE AND OTHER VOLATILE MATTER.**—Weigh accurately from 3 to 5 g of the putty in a tared flat-bottomed dish about 8 cm in diameter, spreading the putty over the bottom. Heat at 105 to 110° C. for one hour, cool, and weigh. Calculate the loss in weight as the percentage of moisture and volatile matter.

(c) **FREEDOM FROM LUMPS OR GRIT.**—Spread a portion of the mixed putty on a clean piece of glass, noting the presence of any lumps. Thin about 5 g of the putty with 3 or 4 cc of raw linseed oil or turpentine, spread the mixture to thin films on the glass, using a wide spatula or putty knife and note the presence of grit.

(d) **WORKING QUALITIES.**—Work up about 10 g of the mixed putty in the hands, noting its tendency to stick. Apply a portion of the putty to the edge of a clean piece of glass, metal, or well-filled wood and work it out to a smooth bevel with a spatula or putty knife. Spread another portion to very thin films on the glass. The putty in both tests shall show good adhesive and elastic properties and shall not be “short” or “mealy.” For comparison purposes a small batch of good putty can be satisfactorily made by taking 20 g of dry, fine natural whiting of known quality, adding about 4 cc of raw linseed oil, and thoroughly kneading to a stiff dough by hand.

(e) **PERCENTAGE OF PIGMENT.**—Weigh accurately about 15 g of the putty in a weighed centrifuge tube. Add 20 to 30 cc of “extraction mixture” (see Reagents), mix thoroughly with a glass rod, wash the rod with more of the extraction mixture, and add enough of the reagent to make a total of 60 cc in the tube. Place the tube in the container of a centrifuge, surround with water, and counterbalance the container of the opposite arm with a similar tube or a tube with water. Whirl at a moderate speed until well settled. Decant the clear supernatant liquid, repeat the extraction twice with 40 cc of extraction mixture and once with 40 cc of ether. After drawing off the ether set the tube in a beaker of water at about 80° C. or on top of a warm oven for 10 minutes, then in an oven at 105 to 110° C. for two hours. Cool, weigh, and calculate the percentage of pigment. Grind the pigment to a fine powder, pass through a No. 80 sieve to remove any skins, and preserve in a stoppered bottle.

(f) **PREPARATION OF FATTY ACIDS.**—To about 25 g of the putty in a porcelain casserole add 15 cc of aqueous sodium hydroxide (see Reagents) and 75 cc of ethyl alcohol, mix and heat uncovered on a steam bath until saponification is complete (about one hour).

Add 100 cc of water, boil, add sulphuric acid of specific gravity 1.2 (8 to 10 cc in excess), boil, stir, and transfer to a separatory funnel to which some water has been previously added. Draw off as much as possible of the acid aqueous layer, wash once with water, than add 50 cc of water and 50 cc of ether. Shake very gently with a whirling action to dissolve the fatty acids in the ether, but not so violently as to form an emulsion. Draw off the aqueous layer and wash the ether layer with one 15 cc portion of water and then with 5 cc portions of water until free from sulphuric acid. Then draw off the water layer completely. Transfer the ether solution to a dry flask and add 25 to 50 g of anhydrous sodium sulphate. Stopper the flask and let stand with occasional shaking at a temperature below 25° C. until the water is completely removed from the ether solution, which will be shown by the solution becoming perfectly clear above the solid sodium sulphate. Decant this clear

solution, if necessary, through a dry filter paper into a dry 100 cc Erlenmeyer flask. Pass a rapid current of dry air (pass through a CaCl_2 tower) into the mouth of the Erlenmeyer flask and heat to a temperature below 75°C . on a dry, hot plate until the ether is entirely driven off.

It is important to follow all of the details, since ether generally contains alcohol, and after washing with water always contains water. It is very difficult to remove water and alcohol by evaporation from fatty acids, but the washing of the ether solution and subsequent drying with anhydrous sodium sulphate removes both water and alcohol. Ether, in the absence of water and alcohol, is easily removed from fatty acids by gentle heat.

The fatty acids prepared as above should be kept in a stoppered flask and examined at once.

(g) TEST FOR MINERAL OIL AND OTHER UNSAPONIFIABLE MATTER.—Place 10 drops of the fatty acid (*f*) in a 50 cc test tube, add 5 cc of alcoholic soda (see Reagents), boil vigorously for five minutes, add 40 cc of water, and mix; a clear solution indicates that not more than traces of unsaponifiable matter are present. If the solution is not clear, the oil is not pure linseed oil.

(h) IODINE NUMBER OF FATTY ACIDS.—Place a small quantity of the fatty acids (*f*) in a small weighing burette or beaker. Weigh accurately. Transfer by dropping about 0.15 g (0.10 to 0.20 g) into a 500 cc bottle having a well-ground glass stopper, or an Erlenmeyer flask having a specially flanged neck for the iodine test. Reweigh the burette or beaker and determine the amount of sample used. Add 10 cc of chloroform. Whirl the bottle to dissolve the sample. Add 10 cc of chloroform to two empty bottles like that used for the sample. Add to each bottle 25 cc of the Hanus solution (see Reagents) and let stand with occasional shaking for one-half hour.

Add 10 cc of the 15 per cent potassium iodide solution and 100 cc of water and titrate with standard sodium thiosulphate, using starch as an indicator. The titrations on the two blank tests should agree within 0.1 cc. From the difference between the average of the blank titrations and the titration on the sample and the iodine value of the thiosulphate solution calculate the iodine number of the sample tested. (Iodine number is centigrams of iodine to 1 g of sample.) If the iodine number is less than 170, the oil does not meet the specification.

(i) COARSE PARTICLES AND SKINS.—Dry in an oven at 105 to 110°C . a No. 325 sieve, cool and weigh accurately. Weigh an amount of putty containing 10 g of pigment (see V, 2, (*e*)), add 100 cc of kerosene, mix thoroughly, and wash with kerosene through the sieve, breaking up all the lumps but not grinding. After wash-

ing with kerosene until all but the particles too coarse to pass the sieve have been washed through wash all kerosene from the sieve with ether or petroleum ether, heat the sieve and contents for one hour at 105 to 110° C., cool and weigh.

3. ANALYSIS OF PIGMENT

(a) QUALITATIVE ANALYSIS.—Make qualitative analysis following ordinary methods.

(b) REACTION AND FREE ALKALI.—Boil 2 g of the pigment for five minutes with 100 cc of water, filter and to the clear filtrate add two drops of phenolphthalein and titrate while hot with 0.1 *N* hydrochloric acid. Not over 0.2 cc of 0.1 *N* HCl shall be required to destroy the red color. The filtrate shall also be neutral to methyl orange indicator.

(c) STRUCTURE.—Compare the structure of the pigment under the microscope with a known specimen of natural (chalk) whiting (with or without the addition of white lead).

(d) CALCIUM CARBONATE (IN THE ABSENCE OF WHITE LEAD).—Weigh accurately 0.25 g of the dry pigment, transfer to a 250 cc beaker, moisten with alcohol, dissolve in about 20 cc of 1 : 1 hydrochloric acid, keeping the beaker covered. Digest for 10 minutes on the steam bath, dilute to about 150 cc, then filter the solution and wash the insoluble residue with hot water. Bring to near boiling and make alkaline with ammonia after adding a few cc of bromine water. Let the precipitate settle, filter, and wash thoroughly with hot water. Take the filtrate and washings from the iron, aluminum, etc., concentrate to about 200 cc, adding a few drops of ammonia. Boil and add while boiling 10 to 15 cc of a hot saturated solution of ammonium oxalate, stir, and continue the boiling until the precipitated calcium oxalate becomes granular. Set on steam bath to settle (about one hour). Filter and wash the oxalate thoroughly with small amounts of hot water. Transfer the moist precipitate to a 400 cc beaker by means of a stream of water from the wash bottle, dissolve the part remaining on the filter by washing with warm dilute sulphuric acid. Add to the beaker 20 cc of sulphuric acid (1 : 1), dilute to about 300 cc with hot water, and titrate the oxalic acid with 0.1 *N* potassium permanganate and report as CaCO₃.¹

$$\begin{aligned} 1 \text{ cc } 0.1 \text{ } N \text{ KMnO}_4 &= 0.0028 \text{ g CaO} \\ &= 0.0050 \text{ g CaCO}_3. \end{aligned}$$

(e) WHITE LEAD AND CALCIUM CARBONATE.—Weigh accurately 1 g of the dry pigment into a 250 cc beaker, moisten with a few drops of alcohol, add slowly 25 to 30 cc of concentrated hydrochloric acid,

¹ If the sample is impure, reference should be made to the methods as given in *The Analysis of Silicate and Carbonate Rocks*, by W. F. Hillebrand, United States Geological Survey Bulletin No. 700.

cover, and boil for 5 to 10 minutes. Dilute to about 150 cc with hot water and heat for about 15 minutes, let settle on the steam bath, filter while hot, and wash any insoluble residue thoroughly with hot water. (Avoid allowing the filter paper to become cold.) Make the solution just alkaline with ammonia, then just faintly acid to litmus, using dilute (1:10) hydrochloric acid. Dilute to about 300 cc and pass hydrogen sulphide gas into the clear solution to complete precipitation. Settle, filter on paper, and wash with water containing some hydrogen sulphide, dissolve the sulphide in hot nitric acid (1:3), add 5 to 10 cc of dilute sulphuric acid (1:1), evaporate until copious fumes of sulphuric anhydride are evolved, cool, add about 75 cc of water, and then about 75 cc of 95 per cent ethyl alcohol. Stir, let settle, filter on a weighed Gooch crucible, wash with dilute alcohol, dry, gently ignite, and weigh as lead sulphate. In the absence of sulphates multiply this weight by the factor 0.854 and report the result as basic carbonate white lead. In the presence of sulphates the weight of lead sulphate multiplied by the factor 0.883 shall be considered white lead. (It is not possible to determine the amount of lead carbonate and lead sulphate when carbonates or sulphates of calcium are present. The arbitrary factor 0.883 is the mean of the largest and smallest of the three factors to convert PbSO_4 to $(\text{PbCO}_3)_2\text{Pb}(\text{OH})_2$, to PbSO_4PbO , and to $(\text{PbSO}_4)_2\text{PbO}$.)

Boil the filtrate from the sulphide separation to expel hydrogen sulphide, add a few drops of nitric acid, and if necessary some ammonium chloride, make alkaline with ammonia, settle, and filter off any precipitate of aluminum, iron, etc., and wash the precipitate with hot water. Proceed to determine total calcium as in V, 3, (d), and report as calcium carbonate.²

4. REAGENTS

(a) STANDARD SODIUM THIOSULPHATE SOLUTION.—Dissolve pure sodium thiosulphate in distilled water (that has been well boiled to free it from carbon dioxide) in the proportion of 24.83 g of crystallized sodium thiosulphate to 1,000 cc of the solution. It is best to let this solution stand for about two weeks before standardizing. Standardize with pure resublimed iodine.³ This solution will be approximately decinormal, and it is best to leave it as it is after determining its exact iodine value rather than to attempt to adjust it to exactly decinormal. Preserve in a stock bottle provided with a guard tube filled with soda lime.

(b) STARCH SOLUTION.—Stir up 2 to 3 g of potato starch or 5 g of soluble starch with 100 cc of 1 per cent salicylic acid solution,

² See footnote 1, p. 6.

³ See Treadwell-Hall, *Analytical Chemistry*, 2, 6th ed., p. 551.

add 300 to 400 cc of boiling water, and boil the mixture until the starch is practically dissolved; then dilute to 1 liter.

(c) **EXTRACTION MIXTURE.**—10 volumes ether (ethyl ether), 6 volumes benzol, 4 volumes methyl alcohol, 1 volume acetone.

(d) **AQUEOUS SODIUM HYDROXIDE.**—Dissolve 100 g of sodium hydroxide in distilled water and dilute to 300 cc.

(e) **POTASSIUM IODIDE SOLUTION.**—Dissolve 150 g of potassium iodide free from iodate in distilled water and dilute to 1,000 cc.

(f) **HANUS SOLUTION.**—Dissolve 13.2 g of iodine in 1,000 cc of 99.5 per cent glacial acetic acid which will not reduce chromic acid. Add enough bromine to double the halogen content determined by titration (3 cc of bromine is about the proper amount). The iodine may be dissolved by the aid of heat, but the solution should be cold when the bromine is added.

(g) **ALCOHOLIC SODIUM HYDROXIDE SOLUTION.**—Dissolve pure sodium hydroxide in 95 per cent ethyl alcohol in the proportion of about 22 g per 1,000 cc. Let stand in a stoppered bottle. Decant the clear liquid into another bottle and keep well stoppered. This solution should be colorless or only slightly yellow when used, and it will keep colorless longer if the alcohol is previously treated with sodium hydroxide (about 80 g to 1,000 cc), kept at about 50° C. for 15 days, and then distilled.

(h) **0.1 N POTASSIUM PERMANGANATE SOLUTION.**—Dissolve 3.161 g of pure potassium permanganate in a liter of distilled water, let stand 8 to 14 days, siphon off the clear solution (or filter through an asbestos filter), and standardize as follows: In a 400 cc beaker dissolve 0.25 to 0.30 g of Bureau of Standards' sodium oxalate in 250 cc of hot water (80 to 90° C.) and add 15 cc of dilute sulphuric acid (1:1). Titrate at once with the potassium permanganate solution, stirring the liquid vigorously and continuously. The permanganate must not be added more rapidly than 10 to 15 cc per minute, and the last 0.5 to 1 cc must be added dropwise, with particular care to allow each drop to be fully decolorized before the next is introduced. The solution should not be below 60° C. by the time the end point is reached. (More rapid cooling may be prevented by allowing the beaker to stand on a small asbestos-covered hot plate during the titration. The use of a small thermometer as a stirring rod is most convenient.) The weight of sodium oxalate used multiplied by 0.7469 gives its CaCO_3 equivalent.

VI. PACKING AND MARKING

No details specified.

VII. ADDITIONAL INFORMATION

This specification covers the requirements for a high-grade prepared putty for general use. It shall be purchased by net weight.

VIII. GENERAL SPECIFICATIONS

No details specified.

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